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Indian Standard

METHODS FOR QUANTITATIVE CHEMICAL ANALYSIS OF BINARY MIXTURES OF SILK AND WOOL OR HAIR

(First Revision)

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NEW DELHI 110002

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Chemical Methods of Test Sectional Committee, TDC 5

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards on 30 December 1988, after the draft finalized by the Chemical Methods of Test Sectional Committee had been approved by the Textile Division Council.

This standard was first published in 1981 and has been revised to include another method based on dissolution of silk in hydrochloric acid as developed by the Textiles Committee, Bombay.

The use of different fibre blends in textiles has necessitated the formulation of standard methods for identification and quantitative estimation of respective fibres. The quantitative analysis of textile fibres in mixtures is of considerable importance to textile technologists, traders and consumers.

While preparing this standard, considerable assistance has been derived from ISO 1833-1977 'Textiles — Binary fibre mixtures — Quantitative chemical analysis', issued by the International Organization for Standardization (ISO).

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHODS FOR QUANTITATIVE CHEMICAL ANALYSIS OF BINARY MIXTURES OF SILK AND WOOL OR HAIR

(First Revision)

1 SCOPE

This standard prescribes two methods for the quantitative chemical analysis of binary mixtures of silk and wool or hair in any textile form, such as fibre, yarn and fabric.

NOTE — Before conducting an analysis according to this standard, the fibres present in the mixture should be identified [*see* IS 667 : 1981 Methods for identification of textile fibres (*first revision*)] and the sample to be analysed should be freed from all non-fibrous matter (*see* IS 9068 : 1979 Recommended methods for the removal of non-fibrous matter prior to quantitative analysis of fibre mixtures). The dye in the dyed fibre is considered to be an integral part of the fibre and is not to be removed.

2 REFERENCES

The Indian Standards listed below are necessary adjuncts to this standard:

IS No.	*Title*
1070 : 1977	Specification for water for general laboratory use (*second revision*)
9068 : 1979	Recommended methods for the removal of non-fibrous matter prior to quantitative analysis of fibre mixtures

3 SAMPLING

3.1 Lot

The quantity of textile material of one definite type and quality delivered to a buyer against one despatch note shall constitute a lot.

3.1.1 If the textile material is fibre or yarn and the lot consists of more than 200 kg of fibre or yarn, it shall be divided into sub-lots, each weighing 200 kg or less.

3.1.2 Each sub-lot shall be tested separately.

3.2 Sampling for Fibre and Yarn

From a sub-lot, 15 increments each weighing approximately 10 g, shall be taken from different parts and mixed thoroughly. This shall constitute a test sample.

3.3 Sampling for Fabrics

3.3.1 The number of pieces to be selected shall be in accordance with Table 1. The pieces thus selected shall constitute a gross sample.

Table 1 Sample Size

(*Clause* 3.3.1)

Lot Size (Number of Pieces)	Sample Size (Number of Pieces)
Up to 100	3
101 ,, 300	4
301 ,, 500	5
501 and above	7

3.3.2 From each piece in the gross sample selected as in **3.3.1**, cut out small portions from at least two different parts weighing about 25 g. The parts selected shall represent the gross sample as far as possible. In the case of fabrics with a definite repetition in weave pattern, the parts selected shall include all yarns in the complete repeat. Dissect small portions of the fabric thus collected into yarn and mix them thoroughly. This shall constitute a test sample.

4 APPARATUS

4.1 Conical Flask — 200 ml minimum capacity, provided with a ground glass stopper.

4.2 Sintered Glass Filter Crucible — appropriate capacity with a pore size of 90 to 150 microns (porosity 1) and provided with a ground glass stopper. If the stopper is not available, the crucible should be enclosed in weighing bottle for weighing.

4.3 Ventilated Oven — capable of maintaining a temperature of 105 ± 3°C.

4.4 Filter Flask — with connection to filter pump and adaptor to enable the crucible (*see* **4.2**) to be fitted to it.

4.5 Analytical Balance — capable of weighing to an accuracy of 0·000 2 g.

4.6 Desiccator — containing self-indicating silica gel or anhydrous calcium chloride.

4.7 Mechanical Shaker

5 REAGENTS

5.1 Quality of Reagents

Unless specified otherwise, pure chemicals shall be employed in tests and distilled water (*see* IS 1070 : 1977) shall be used where the use of water as a reagent is intended.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the test results.

5.2 Dilute Sulphuric Acid Solution — 5 percent (*m*/*m*).

5.3 Sulphuric Acid Solution — 75 percent (*m*/*m*).

5.4 Dilute Ammonia Solution — 20 percent (*v*/*v*).

5.5 Hydrochloric Acid Solution — 28 percent (*m*/*m*).

5.6 Dilute Hydrochloric Acid Solution — 5 percent (*m*/*m*).

6 TEST CONDITIONS

6.1 The test shall be conducted in prevailing atmospheric conditions.

NOTE — Since dry masses are determined, it is not necessary to condition the sample.

7 PREPARATION OF TEST SPECIMENS

From the sample, after removing size and finishes as recommended in IS 9068 : 1979, draw a representative sample weighing about 2 to 3 g. Cut the yarn into pieces and dissect the cloth into yarn pieces of about 10 mm length.

8 METHOD 1

8.1 Principle

The silk fibre is dissolved from a known dry mass of the mixture with 75 percent (*m*/*m*) sulphuric acid (*see* Note). The residue is collected, washed, dried and weighed; its mass corrected if necessary, and is expressed as a percentage of the dry mass of the mixture. The percentage of silk is found by difference.

NOTE — Wild silk, such as tussah, does not completely dissolve in 75 percent (*m*/*m*) sulphuric acid.

8.2 Procedure

8.2.1 Take a specimen weighing about 1 g from the pretreated sample (*see* 7). Dry the pieces in weighing bottle at 105 ± 3°C to constant mass, cool it in a desiccator and obtain the oven dry mass of the specimen.

NOTE — The mass shall be taken as constant if the difference between any two successive weighings at an interval of 20 minutes does not exceed 0·1 percent.

8.2.2 Put the specimen in a glass-stoppered conical flask. Add 100 ml of sulphuric acid solution per gram of the specimen, insert the stopper, shake vigorously (preferably in a mechanical shaker) and allow to stand for 30 minutes at room temperature. Shake again and allow to stand for 30 minutes. Shake for the last time and filter the contents of the flask through the weighed filter crucible. Wash any remaining fibres from the flask with a little sulphuric acid solution. Drain the crucible by suction and wash the residue on the crucible successively with 50 ml each of dilute sulphuric acid solution, water and dilute ammonia solution. Each time, allow the fibres to remain in contact with the liquid for about 10 minutes before applying suction. Finally, rinse with water, leaving the fibres in contact with water for about 30 minutes. Drain the crucible by suction. Dry the crucible and residue at 105 ± 3°C temperature, and cool and weigh.

8.2.3 Repeat the procedure prescribed in **8.2.1** and **8.2.2** with the remaining test specimen(s).

8.3 Calculations

8.3.1 *Method Based on Clean Dry Mass*

Calculate the percentage (*P*) of clean dry insoluble component by the formula:

$$P = \frac{100 \times m_1 \times d}{m_0}$$

where

m_0 = dry mass of the specimen,

m_1 = dry mass of the residue, and

d = correction factor of variation in mass of the insoluble component in the reagent.

NOTE — The value of *d* is found to be 0·975.

8.3.2 *Method Based on Clean Dry Mass with Percentage Additions for Moisture*

Calculate the percentage (P_M) of clean insoluble component with percentage additions for moisture, by the formula:

$$P_M = \frac{100 \times P \times \left[1 + \frac{b}{100}\right]}{P\left[1 + \frac{b}{100}\right] + (100 - P)\left[1 + \frac{a}{100}\right]}$$

where

P_M = percentage, by mass, of wool or hair fibre in the test sample on dry mass with moisture allowance,

P = percentage of clean dry insoluble component,

a = percentage addition for moisture to the soluble component, and

b = percentage addition for moisture to the insoluble component.

NOTES

1 The following values for standard moisture regain of various fibres may be considered:

Fibre	*Standard Moisture Regain (Percent)*
Silk	11·0
Wool	13·6

2 The standard moisture regain values are generally accepted as commercial moisture regain values in the trade.

8.3.3 *Method Based on Clean Dry Mass with Percentage Additions for Moisture and Non-Fibrous Matter*

Calculate the percentage (P_A) of clean insoluble component in the mixture with percentage additions for moisture and non-fibrous matter by the following formula (*see also* Notes 1 and 2 under **8.3.2**):

$$P_A = \frac{100 \times P \times \left[1 + \frac{a_2 + b_2}{100}\right]}{P \times \left[1 + \frac{a_2 + b_2}{100}\right] + (100 - P)\left[1 + \frac{a_1 + b_1}{100}\right]}$$

where

P = percentage of clean dry insoluble component,

a_1 = percentage addition for moisture to the soluble component,

a_2 = percentage addition for moisture to the insoluble component,

b_1 = percentage addition for non-fibrous matter to the soluble component, and

b_2 = percentage addition for non-fibrous matter to the insoluble component.

NOTE — The percentage additions for non-fibrous matter may be as agreed to between the buyer and the seller.

8.4 Determine the percentage of silk fibre by difference.

9 METHOD 2

9.1 Principle

The silk fibre is dissolved from a known dry mass of the mixture with 28 percent (*m/m*) hydrochloric acid. The residue is collected, washed, dried and weighed; its mass corrected, if necessary, and is expressed as a percentage of the dry mass of the mixture. The percentage of silk is found by difference.

9.2 Procedure

9.2.1 Follow the procedure given in **8.2.1**.

9.2.2 Put the specimen in a glass-stoppered conical flask. Add 100 ml of hydrochloric acid solution per gram of the specimen, insert the stopper, shake vigorously (preferably in a mechanical shaker) and allow to stand at room temperature for 30 minutes. Shake again and allow to stand for 30 minutes. Shake for the last time and filter the contents of the flask through the weighed filter crucible. Wash any remaining fibres from the flask with a little hydrochloric acid solution. Drain the crucible by suction and wash the residue on the crucible successively with 50 ml each of dilute hydrochloric acid solution, water and dilute ammonia solution. Each time, allow the fibres to remain in contact with the liquid for about 10 minutes before applying suction. Finally, rinse with water, leaving the fibres in contact with the water for about 30 minutes. Drain the crucible by suction. Dry the crucible and residue at 105 ± 3°C temperature, and cool and weigh.

9.2.3 Repeat the procedure prescribed in **9.2.1** and **9.2.2** with the remaining test specimen(s).

9.3 Calculations

Calculate the percent of component fibres in the mixture as prescribed in **8.3.1**, **8.3.2**, **8.3.3** and **8.4** taking the value of correction factor *d* as 1·00.

10 REPORT

The report shall include the following:

a) Nature of material to be tested,

b) Method used (*see* **8** or **9**),

c) Method of calculation used (*see* **8.3** and **9.3**),

d) Number of specimens tested, and

e) Percentage of component fibres in the mixture (individual and average).

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